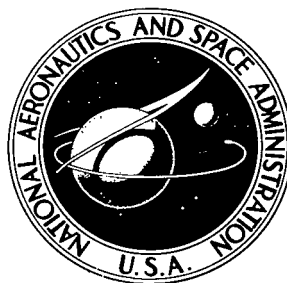


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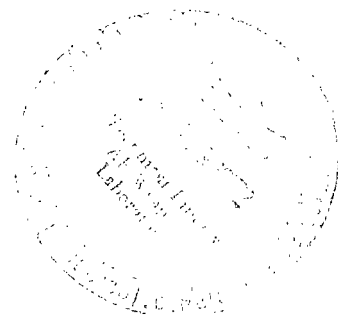
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ACHIEVEMENT OF A LOW-OUTGASSING WHITE PAINT SYSTEM FOR SPACECRAFT THERMAL CONTROL

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16. Abstract Test results and data for achieving a low-outgassing polymer resin suitable for potting or as a vehicle for a paint pigment are presented. The resin, prepared in 0.5-kg (1-lb) batches, is acceptable for spacecraft use; its weight loss is less than 0.5 percent and the volatile condensable materials are less than 0.05 percent. The paint adheres well to a primed fiber glass or aluminum substrate. Results of UV irradiation, electron and proton radiation, and thermal cycling are presented.					
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ACHIEVEMENT OF A LOW-OUTGASSING WHITE PAINT SYSTEM FOR SPACECRAFT THERMAL CONTROL

by

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Goddard Space Flight Center

INTRODUCTION

Thermal control of spacecraft has necessitated the judicious use of thermal control coatings having specific ratios of absorptance α to emittance ϵ . These coatings have included numerous white or black paints and even certain reflecting tapes. One important disadvantage of paints is their high outgassing, necessitated in part by the use of volatile solvents for spray applications. Of the temperature control coatings tested by Stanford Research Institute (SRI) for outgassing (Reference 1), not one of them could pass both the total weight loss maximum of 1 percent and the volatile condensable materials maximum of 0.1 percent. Thus, the evaporation and condensation of these volatiles in a thermal/vacuum environment or in the space environment was highly probable.

The important consideration of paint's outgassing is obvious when one considers the total area covered by paint in a satellite. The Nimbus E spacecraft has an approximate total surface area of 187 000 cm² (29 000 in²), and of this almost 16 percent or 29 700 cm² (4600 in²) is covered by the PV-100¹ white paint. The PV-100 is an alkyd-modified silicone pigmented with titanium dioxide; it has a high outgassing profile even though it is cured at an elevated temperature. Specifically, the data for the PV-100 from the SRI report indicate a 2.48 percent weight loss after a room temperature cure and 1.51 percent weight loss after a 423 K (150° C) cure for 24 hr.

This report describes the preparation and the properties of a low-outgassing vehicle that can be used as a potting compound or as the resin in a white or black paint. The paint mixture has less than 1 percent total weight loss and less than 0.1 percent volatile condensables when applied by spraying, even though it requires only a room temperature cure. Methods of application have been worked out and tests of its adhesion to aluminum and to epoxy fiber glass have been performed successfully.

¹A product of Vita Var Corp.

OUTGASSING OF DEVOLATILIZED RESIN

Outgassing Test

Numerous outgassing tests have been conducted at Goddard Space Flight Center. These tests have indicated that a considerable portion of the outgassing occurs within the first few hours in a thermal/vacuum environment with the loss principally of low molecular weight compounds. This observation led to the consideration of devolatilizing certain polymers as a preliminary step that would hopefully reduce the outgassing of the final mixture without radically affecting its desirable properties. Once devolatilized, the mixture would be tested in the standardized outgassing apparatus.

The Micro-VCM apparatus² is practically identical to that employed at SRI (Reference 1). The sample is heated to 398 K (125° C) for 24 hr in a vacuum of about 1×10^{-4} N/m² (1×10^{-6} torr) and its weight loss is determined from the measured initial and final weights. The volatiles from the sample impinge upon a collector plate that is at 298 K (25° C) to permit condensation, with the measured initial and final weights of the collector plate permitting the calculation of the volatile condensable materials (VCM). As a preliminary step in the outgassing measurements, each sample is conditioned for 24 hr in a 50 percent relative humidity atmosphere at room temperature; this reduces the variable of a dry versus wet laboratory atmosphere and starts all materials from the same baseline. The evaporation and condensation occur within the vacuum chamber and during the full 24 hr of the test. The criteria for acceptability of a material from the outgassing standpoint are maxima of 1 percent weight loss and 0.1 percent VCM. This particular test succeeds in screening out those potentially dangerous materials, i.e., dangerous to optical experiments in their absorption of radiation in specific wavelength regions.

Devolatilized Resin

The candidate selected for devolatilization following a few successful preliminary steps was RTV-602,³ a silicone used frequently as a potting compound. The RTV-602, mixed with zinc oxide, has been applied as a paint to the back of the solar paddles on the OAO C spacecraft. It has numerous uses as a potting compound on other spacecraft components.

The preliminary steps involved the thermal/vacuum treatment of small amounts of RTV-602. The variations involved were in temperature, vacuum, container, and/or height of fluid in the container. The devolatilized resin was then mixed with a catalyst, cured, and later tested in the Micro-VCM apparatus. The preliminary results of the Micro-VCM tests clearly showed the effectiveness of a 423 K (150° C) treatment for 24 hr in a good vacuum and with a resin height of about 12 mm ($\sim\frac{1}{2}$ in.). The effect of resin height was noted in the use of a narrow glass tube in which the fluid height was approximately 50 mm (~ 2 in.) but which had an excessive weight loss in the outgassing test.

The upgrading of the technique consisted of the devolatilization of 0.5-kg (1-lb) lots of RTV-602. The total time was set at 24 hr, the temperature at 423 ± 3 K ($150^\circ \pm 3^\circ$ C), and the vacuum on the

²A. Fisher and B. Mermelstein: "GSFC Micro-Volatile Condensable Materials System for Polymer Outgassing Studies." NASA Document X-735-69-471, Oct. 1969.

³A product of the General Electric Co.

order of $7 \times 10^{-4} \text{ N/m}^2$ (5×10^{-6} torr). The vacuum is an average value, decreasing from 7×10^{-3} to about $7 \times 10^{-5} \text{ N/m}^2$ (5×10^{-5} to about 5×10^{-7} torr) during the bakeout.

The selection of the 423 K (150° C) temperature was determined with the aid of carefully monitored tests on small-volume samples in which the variation in temperature was reduced to $\pm 2 \text{ K}$ ($\pm 2 \text{ C}^\circ$). The larger batches were treated in a separate vacuum chamber in which the temperature control was less exact. There were three separate thermocouples immersed in the silicone resin and the average of these thermocouple readings was used for adjusting the temperature. The consequent temperatures at an individual thermocouple location may have been between a low of 401 K (128° C) in batch 6 to a high of 443 K (170° C) in batch 8. The computed average temperature for the 24-hr bake of each accepted batch and the initial and final vacuum readings are given in Table 1.

Outgassing Test Results

Preliminary results had shown the feasibility of devolatilization as an effective technique. The outgassing tests were run shortly after the bakeout as a means of keeping track of the worth of the method.

A relatively small portion of the P-764 (devolatilized RTV-602) was mixed with the catalyst⁴ (0.25 percent), cured for 7 days at room temperature, and then tested in the Micro-VCM apparatus. The outgassing results for P-764 are presented in Table 2. Each of the devolatilized specimens had a very low total weight loss and a very low VCM, well below the maxima suggested for spacecraft use.

An additional measurement was taken of the viscosity before and after the devolatilization as a possible aid in estimating the outgassing characteristics of the silicone. As might be expected, a significant change in viscosity occurred during the devolatilization step; the viscosity of the starting

Table 1—Devolatilization conditions.

Batch	Average Devolatilization Temperatures for 24-hr Periods (K (°C))	Vacuum at 24 hr (N/m ² (torr))	Vacuum at 1 hr (N/m ² (torr))
2	416.8 (143.6)	3.1×10^{-5} (2.4×10^{-7})	2.2×10^{-4} (1.7×10^{-6})
4	421.0 (147.8)	1.4×10^{-5} (1.1×10^{-7})	1.7×10^{-4} (1.3×10^{-6})
5	411.3 (138.1)	2.1×10^{-5} (1.6×10^{-7})	1.4×10^{-4} (1.1×10^{-6})
6	412.0 (138.8)	2.5×10^{-5} (1.9×10^{-7})	1.2×10^{-2} (9.0×10^{-5})
7	426.8 (153.6)	2.6×10^{-4} (2.0×10^{-6})	5.6×10^{-3} (4.3×10^{-5})
8	426.3 (153.1)	8.8×10^{-5} (6.8×10^{-7})	9.1×10^{-3} (7.0×10^{-5})
9	429.9 (156.7)	2.0×10^{-5} (1.5×10^{-7})	1.8×10^{-4} (1.4×10^{-6})
10	432.1 (158.9)	1.3×10^{-5} (1.0×10^{-7})	7.8×10^{-3} (6.0×10^{-5})
11	429.5 (156.3)	4.6×10^{-5} (3.5×10^{-8})	1.0×10^{-2} (8.0×10^{-5})

⁴SRC-05 catalyst from the General Electric Co. was used.

Table 2—Outgassing results for P-764.

Batch No.	Weight Loss ^a (percent)	VCM ^a (percent)	Viscosity After Devolatilization (N-s/m ²)
2	0.370	0.055	1.820
4	.305	.000	2.272
5	.385	.035	1.584
6	.370	.035	1.552
7	.350	.020	2.024
8	.370	.005	2.064
9	.404	.012	2.256
10	.335	.015	2.740
11	.315	.009	2.280

^aAverage of duplicate tests.

resin was 1.065 ± 0.025 N-s/m² (1065 ± 25 cP) but the final viscosity was more variable and attributable to temperature variations during the heating. No significant correlation between weight loss and final viscosity was noted.

Catalyst Content Versus Pot Life

When using the P-764 with the recommended amount of catalyst for the RTV-602, a speedier reaction and a much shorter pot life resulted. Gel time measurements were made for 20-g samples with different amounts of catalyst and are as follows:

- (1) 0.25 percent catalyst: gel times of 27.2 and 33.6 min
- (2) 0.50 percent catalyst: gel time of 3.9 min

The selection of 0.25 percent catalyst is based upon the desire for a reasonable time before having to pour or apply the mixture. Comparisons of P-764 and RTV-602 with 0.25 percent catalyst are listed as follows:

- (1) P-764: 15- to 20-min pot life and a hardness of 20 Shore A
- (2) RTV-602 (from manufacturer's literature): 2- to 3-hr pot life and a hardness of 15 Shore A

PAINT DEVELOPMENT

Preparation and Outgassing Test

The low outgassing of the P-764 and the knowledge that a combination of RTV-602 and ZnO had been used as a paint led to the development of the P-764-1 paint. The procedure used as a guide was

that prepared by Grumman Aerospace Corp. personnel for the OAO program. The manufacturer's analysis of the zinc oxide pigment⁵ is given in Table 3. The physical properties are—

- (1) Specific gravity = 5.6.
- (2) Specific surface = 3.0 to 4.3 m²/g.
- (3) Surface mean diameter = 0.25 to 0.35 μ m.

Relatively small amounts of paint were prepared. The proportions by weight of the constituents included 100 parts P-764, 240 parts zinc oxide, 1 part catalyst, and 200 parts toluene. The amount of catalyst was increased to 1 percent of the P-764, and the amount of toluene was sufficient to make the paint sprayable. The technique for mixing these small paint formulations is given in the appendix.

A number of paint samples, now identified as P-764-1, were sprayed onto substrates, cured for 7 days at room temperature, and then tested for outgassing. Obviously, the use of a volatile solvent is likely to increase the outgassing due to entrapped solvent; the Micro-VCM test must be used to confirm the outgassing regardless of the low-outgassing rate of the individual paint components. The results of the P-764-1 outgassing tests on paints made from batch 4 are listed in Table 4. These results are extremely encouraging, particularly because both the total weight loss and the VCM are below the respective maxima of 1 and 0.10 percent.

Table 3—Representative chemical analysis of ZnO pigment.

Component	Percent	Component	Percent
ZnO	99.99	CdO	0.0001
SO ₃	.001	Al	<.001
Fe	.0001	Ca	<.001
Cu	<.0001	Na	<.001
Mn	<.0001	Si	<.0005
PbO	.0006	Mg	<.0004
As ₂ O ₃	.00001		

Table 4—Outgassing results from batch 4 paints and resin.

Sample	Total Weight Loss (percent)	VCM (percent)
P-764-1, matte finish	0.278	0.011
P-764-1, glossy finish	.331	.006
P-764, resin	.305	.000

⁵S.P. ZnO-500, Lot 781, was used as obtained from the New Jersey Zinc Co.

A gel time of the P-764-1 paint, using a 30-g sample and 1.0 percent catalyst, was also determined. Gel times of 3 hr 34.5 min and 3 hr 50.8 min were obtained. It is concluded that paint mixtures are usable for over 2 hr, depending in part upon the ambient temperature. It is recommended that small amounts of paint be catalyzed to determine the gel time before the desirable amount of paint is prepared for the intended area of application.

Irradiation Test

Tests were also conducted to determine the effect of ultraviolet, electron, and proton irradiation on the paint. From previous experience with thermal control paints, there was little expectation that the P-764-1 would withstand the effects of ultraviolet light or of protons. The hope was that P-764-1 would withstand the effects better than the RTV-602 plus ZnO did, or at least no worse. The paints were prepared as before and sprayed onto 2.5-cm (1-in.) diameter aluminum discs.

Degradation was measured by comparing these specimens.⁶ The irradiation source was a solar simulator⁷ that was set for 1 equivalent UV Sun. Measurements of the paint's reflectance were made in situ with a spectrophotometer.⁸ In the first hour, the temperature of the aluminum sample increased from 292 to 353 K (19° to 80° C) because of a poor thermal contact with the cooling plate; after improvements, the sample was maintained at ~300 K (~27° C) during the remainder of the test.

The samples received 165 equivalent UV Sun hours in vacuum. The solar absorptance was computed and a comparison was made with the initial solar absorptance. The computed values are presented in Table 5 and resultant curves are presented in Figures 1 and 2. It is interesting to note that the P-764-1 paint with the matte appearance had a change of 39 percent in solar absorptance equal to that of the RTV-602 paint. The relatively small change of 13 percent for the glossy P-764-1 paint is not presently explainable; other samples will be tested to determine whether the change is reproducible.

It should be pointed out that a white silicone paint is presently being tested that should have a resistance to UV irradiation that is superior to that of P-764-1. This will be identified as P-764-1-A. In essence this paint utilizes P-764 silicone resin and a buffered zinc oxide pigment that is coated with potassium silicate.⁹ Its resistance to UV irradiation should be very similar to that of IITRI's white thermal control paint (References 2 to 4).

Table 5—Computed solar absorptance.

Condition	RTV-602 Paint	P-764-1	
		Matte	Glossy
Preirradiation (in vacuum)	19.2	18.4	19.4
After 165 hr	26.7	25.7	22.0
Change	7.5	7.3	2.6
Percent change	39	39	13

⁶R. McIntosh, J. Bass, and J. Mann, private communication, Apr. 27, 1971.

⁷A Spectrolab X-25 solar simulator was used.

⁸A Beckman DK-2A spectrophotometer was used.

⁹This pigment was received from IIT Research Institute, Chicago.

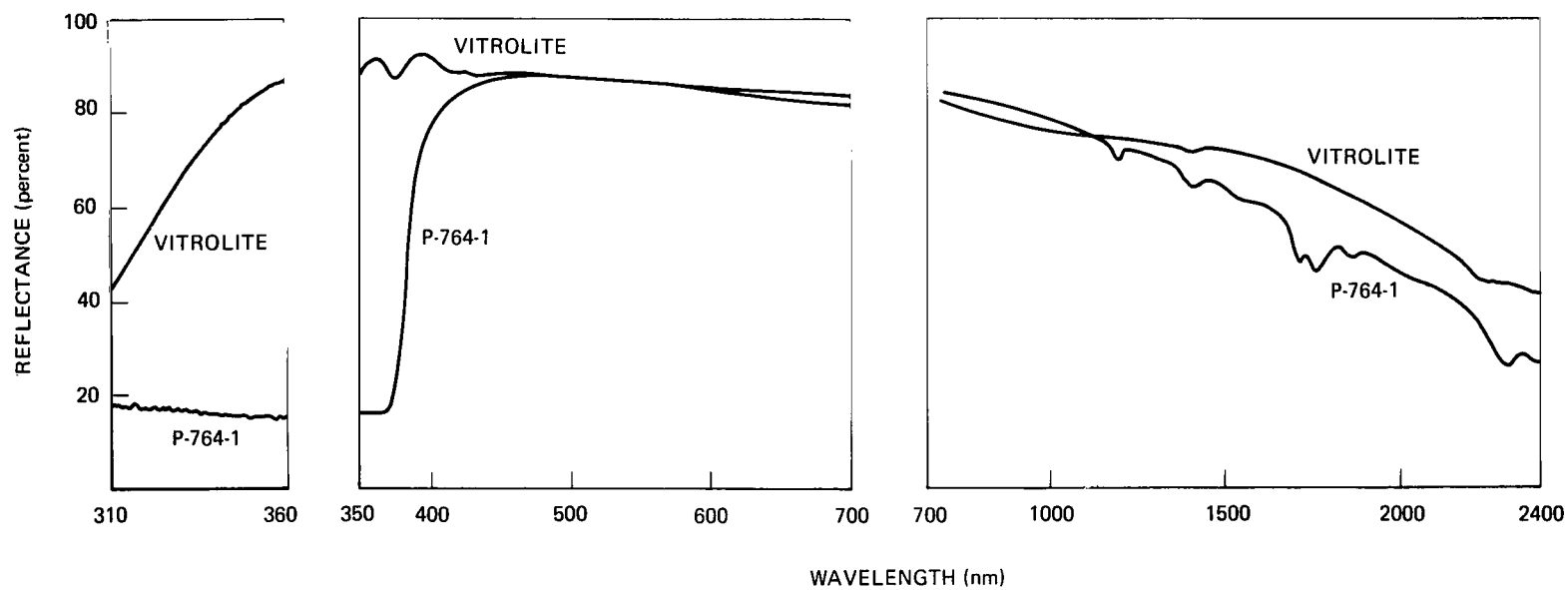


Figure 1—Reflectance of P-764-1, glossy finish, after 165 equivalent UV Sun hours. Vitrolite (registered trademark of Drano Corp., Foxboro, Mass.) is given as a reference material.

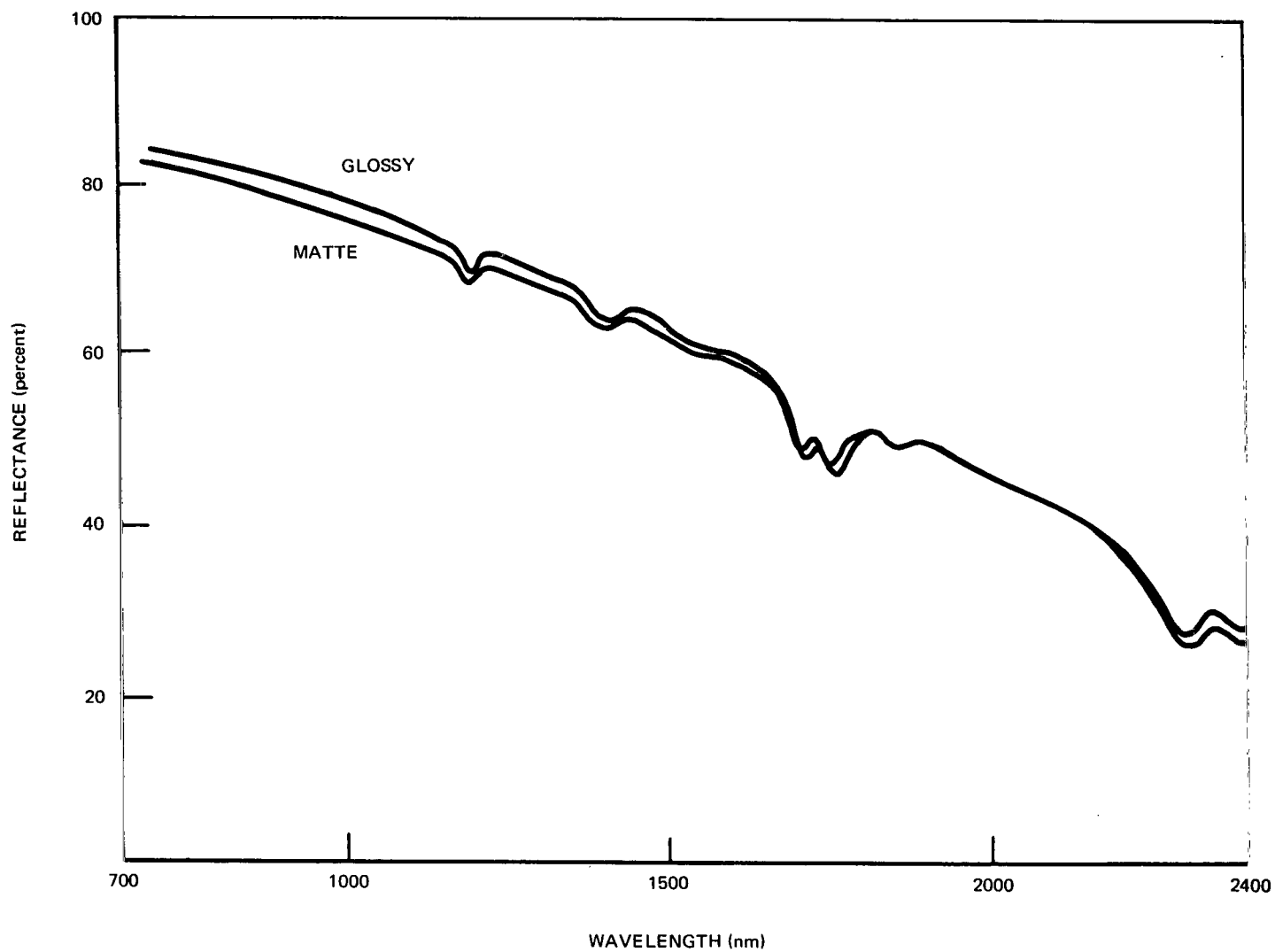


Figure 2—Comparison of reflectance in the infrared region of glossy and matte finishes of P-764-1 paint after 165 equivalent UV Sun hours.

The effect of electrons and protons on the paint reflectance was determined by subjecting samples to estimated 1- and 2-yr exposures for the ATS spacecraft. The electron flux was 5×10^{10} electrons/s/cm² at 2.4×10^{-13} J (1.5 MeV) and the proton flux was 1×10^{12} protons/s/cm² at 2.4×10^{-13} J. The total exposures for the 2-yr level were 1×10^{13} electrons/cm² and 1×10^{15} protons/cm² while under a vacuum of about 1.1×10^{-3} N/m² (8×10^{-6} torr).

The effect of electron irradiation upon all samples resulted in negligible changes; but the proton irradiation caused significant changes, as expected. The reflectance curves for proton-irradiated samples are presented in Figure 3. The comparison of these curves shows that each paint degraded in the visible region in comparison to the unirradiated RTV-602 + ZnO. Each sample was also compared to a reference (not shown) located generally at 100 percent and obtained from the reflectance of an MgO standard in air. It is noteworthy that both of the P-764-1 paints showed an improvement in the infra-red region (Figure 3).

Thermal Cycling

Paint test samples were prepared on aluminum (6061) and on epoxy fiber glass plastic substrates. The surfaces had been prepared in three different ways: (1) no treatment, (2) primer only, and (3) roughening the surface plus primer. The primer¹⁰ was applied by brush to a thickness of about 6.3 μ m (0.25 mil). (This thickness is the upper limit for good adhesion of paint to substrate.) Temperature cycles went from 383 to 203 K (110° to -70° C); there were 30 cycles, 1 cycle/hr, with samples held at the hot or cold temperature approximately 25 min. Visual examination of the cycled paint samples did not reveal any blistering, cracking, or flaking. No visible change occurred; however, the unprimed samples could be pried up easily though no tearing occurred. Paint samples having primed substrates showed excellent adhesion; the paint could only be removed by cutting with a scalpel; it did not lift easily from the substrate.

SUMMARY

The P-764-1 paint is recommended for use as a thermal control coating because of its ease of application, its low outgassing, and its excellent adhesion. It is noteworthy, too, that P-764 is an excellent potting compound and is superior to the original RTV silicone resin in its outgassing characteristics.

The technique of devolatilization of a polymer is thus shown to be significant in reducing outgassing. However, the effect may be dependent upon the polymeric structure; such a dependence would require sufficient testing to achieve suitable temperature, vacuum, and time parameters for devolatilization of other polymers.

¹⁰SS4155 primer from the General Electric Co. was used.

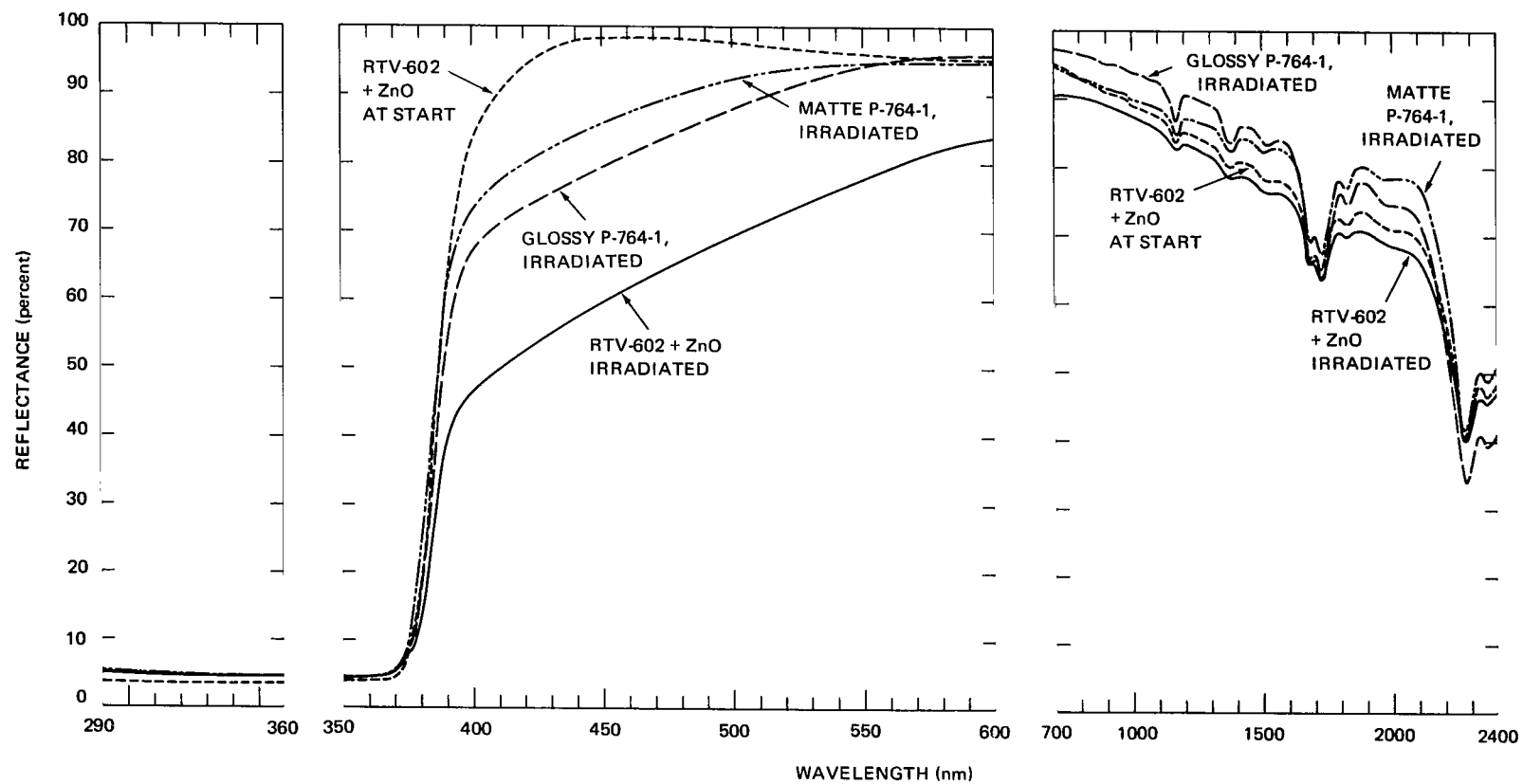


Figure 3—Reflectance of paint samples after equivalent of 2-yr exposure on ATS spacecraft (proton irradiation of about 1×10^{15} protons/cm² at a flux level of 1×10^{12} protons/s/cm² at 2.4×10^{-13} J (1.5 MeV)).

ACKNOWLEDGMENTS

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Goddard Space Flight Center
National Aeronautics and Space Administration
Greenbelt, Maryland, January 21, 1972
831-41-75-01-51

Appendix

P-764 PAINT DEVELOPMENT PROGRAM

PAINT FORMULATION AND SOURCES OF CONSTITUENTS

- (1) P-764 silicone, 5.0 g, prepared by devolatilization of RTV-602 silicone for 24 hr at 423 K (150° C) in vacuum of 7×10^{-4} N/m² ($\sim 5 \times 10^{-6}$ torr)
- (2) Zinc oxide (SP-500 from the New Jersey Zinc Co.), 12.0 g, air dried 48 hr at 398 K (125° C) prior to use; not screened after drying
- (3) SRC-05 catalyst, 0.050 g, as obtained from General Electric with the RTV-602.
- (4) Toluene, 10.0 g, certified grade.

PAINT MIXING PROCEDURE

- (1) 5.0 g of RTV-602 silicone were placed into mortar.
- (2) Approximately one-third of the ZnO was added to the silicone.
- (3) With a pestle, the components were mixed until uniform.
- (4) When the zinc oxide powder had blended into the RTV-602, another third of the powder was added and mixed well with the pestle.
- (5) The remainder of zinc oxide was added and the mixing was continued until the zinc oxide and silicone paste became smooth.
- (6) The mixture was transferred to a sprayer jar and the 10.0 g of solvent was added. This mixture was blended until the silicone/zinc oxide paste was well dispersed into the solvent.
- (7) Immediately prior to the application of the paint mix, 0.050 g of catalyst was added and well blended.
- (8) Paint was sprayed with 1.0×10^5 N/m² (15 psi) dry nitrogen until a sufficient paint coating was obtained. A crosscoat type of application was used to obtain a uniform thickness. A Passche model AU airbrush gun was used to spray paint.
- (9) The paint was air dried for 7 days at ambient temperature for total cure.

SUBSTRATE SURFACE PREPARATION

The following surface preparation was used on both aluminum and fiber glass surfaces. Only the thermal cycling substrate material was given a surface preparation. All other surfaces were solvent wiped only.

- (1) The surface was wiped clean with toluene and then with ethyl alcohol (200 proof).
- (2) The surface was lightly sanded with a 220-grit SiC paper.
- (3) Grit particles were removed by wiping with ethyl alcohol.
- (4) The surface was blown dry with pressurized Freon 12.

The following method of priming was used for both aluminum and fiber glass surfaces.

- (1) The GE SS4155 primer was applied by brush to obtain a thin uniform coating that was not to exceed $6.3\ \mu\text{m}$ (0.25 mil). (A spray technique could have been used.)
- (2) The primer coat was air dried at ambient temperature for a minimum of 1 hr before applying the paint coating.

(All fresh paint samples were prepared using 1.0 percent catalyst; less catalyst would have been used for paint batches that were not freshly made.)

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